PB88-154265

Direct/Delayed Response Project: Field Operations and Quality Assurance Report for Soil Sampling and Preparation in the Southern Blue Ridge Province of the United States. Volume 2. Preparation

Lockheed Engineering and Management Services Co., Inc., Las Vegas, NV

Prepared for

Environmental Monitoring Systems Lab.
Las Vegas, NV

Jan 88

U.S. Department of Commerce
National Technical Information Service

PB88-154265

EPA/600/4-87/041b
January 1988

DIRECT / DELAYED RESPONSE PROJECT: FIELD OPERATIONS AND QUALITY ASSURANCE REPORT FOR SOIL SAMPLING AND PREPARATION IN THE SOUTHERN BLUE RIDGE PROVINCE OF THE UNITED STATES

VOLUME II PREPARATION

by

M. F. Haren and R. D. Van Remortel
Lockheed Engineering and Management Services Company, Inc.
Las Vegas, Nevada 89119

Contract Number 68-03-3249

Project Officer

L. J. Blume

Exposure Assessment Research Division
Environmental Monitoring Systems Laboratory
Las Vegas, Nevada 89193-3478

ENVIRONMENTAL MONITORING SYSTEMS LABORATORY
OFFICE OF RESEARCH AND DEVELOPMENT
U. S. ENVIRONMENTAL PROTECTION AGENCY
LAS VEGAS, NEVADA 89193-3478

REPRODUCED BY
U.S. DEPARTMENT OF COMMERCE
NATIONAL TECHNICAL
INFORMATION SERVICE
SPRINGFIELD, VA. 22161

TECHNICAL REPORT DATA
(Please read Instructions on the reverse before completing)

1. REPORT NO. EPA/600/4-87/041b	2.
3. RECIPIENT'S ACCESSION NO. PB88 154265/AS	
4. TITLE AND SUBTITLE DIRECT/DELAYED RESPONSE PROJECT: FIELD OPERATIONS AND QUALITY ASSURANCE REPORT FOR SOIL SAMPLING AND PREPARATION IN THE SOUTHERN BLUE RIDGE PROVINCE OF THE UNITED STATES VOLUME II PREPARATION	5. REPORT DATE January 1987 6. PERFORMING ORGANIZATION CODE
7. AUTHOR(S) M. F. Haren and R. D. Van Remortel	8. PERFORMING ORGANIZATION REPORT NO.
9. PERFORMING ORGANIZATION NAME AND ADDRESS Lockheed Engineering and Management Services Co., Inc. Las Vegas, NV 89119	10. PROGRAM ELEMENT NO. 11. CONTRACT/GRANT NO. 68-03-3249
12. SPONSORING AGENCY NAME AND ADDRESS Environmental Monitoring Systems Laboratory, LV, NV Office of Research and Development U.S. Environmental Protection Agency Las Vegas, NV 89193-3478	13. TYPE OF REPORT AND PERIOD COVERED 14. SPONSORING AGENCY CODE EPA/600/07
15. SUPPLEMENTARY NOTES	

16. ABSTRACT

The Direct/Delayed Response Project Soil Survey includes the mapping, characterization, sampling, preparation, and analysis of soils in order to assess watershed response to acidic deposition within various regions of the United States. Soil samples collected by sampling crews in the Southern Blue Ridge Province were processed at two preparation laboratories before being sent for analysis at four analytical laboratories. Volumes I and II summarize the procedural and operational compliance with protocols used by the sampling crews and by the preparation laboratories, respectively. Deviations from protocols and difficulties encountered are identified and discussed. Recommendations are made for program improvement.

In general, soil sampling activities during the survey proceeded as planned. A review of the soil data suggests that the integrity of the soil samples was maintained during the preparation activities. In most cases, sampling crews and laboratory personnel adhered to protocols.

17. KEY WORDS AND DOCUMENT ANALYSIS

a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group

18. DISTRIBUTION STATEMENT RELEASE TO PUBLIC	19. SECURITY CLASS *(This Report)* UNCLASSIFIED	21. NO. OF PAGES 78
	20. SECURITY CLASS *(This page)* UNCLASSIFIED	22. PRICE

EPA Form 2220-1 (Rev. 4-77) PREVIOUS EDITION IS OBSOLETE

NOTICE

The information in this document has been funded wholly or in part by the United States Environmental Protection Agency under contract number 68-03-3249 to Lockheed Engineering and Management Services Company, Inc. It has been subject to the Agency's peer and administrative review, and it has been approved for publication as an EPA document. The mention of trade names or commercial products does not constitute endorsement or recommendation for use.

ABSTRACT

The Direct/Delayed Response Project Soil Survey includes the mapping, characterization, sampling, preparation, and analysis of soils in order to assess watershed response to acidic deposition within various regions of the United States. Soil samples from the Southern Blue Ridge Province were transported to preparation laboratories for processing before delivery to analytical laboratories. This document summarizes procedural and operational compliance with the protocols used at the preparation laboratories. Deviations from the protocols and difficulties encountered are identified and discussed. Recommendations are made for program improvement.

A review of the soil data suggests that the integrity of the soil samples was maintained during the preparation activities. In most cases, laboratory personnel adhered to protocols.

This report was submitted in partial fulfillment of contract number 68-03-3249 by Lockheed Engineering and Management Services Company, Inc. under the sponsorship of the U.S. Environmental Protection Agency. The report covers a period from March, 1986 to December, 1986, and work was completed as of October, 1987.

CONTENTS

Page

Notice ii
Abstract iii
Figures vi
Tables vi
Acknowledgments vii

1. Introduction 1
Overview 1
Objectives 3

2. Sample Preparation Methods and Analysis 5
Sample drying 5
Moisture determination 5
Crushing and sieving 5
Rock fragment determination 5
Soil homogenization 6
Qualitative test for inorganic carbon 6
Bulk density determination 8

3. Preparation Laboratory Operations 8
Sample receipt and storage 9
Equipment inventory 9
Sample drying 9
Moisture determination 10
Crushing and sieving 10
Rock fragment determination 10
Soil homogenization 10
Qualitative test for inorganic carbon 10
Bulk density determination 11
Sample shipment 11
Record keeping 13

4. Quality Assurance/Quality Control 13
Design components 13
Training 13
Communications 13
Data quality objectives 15
On-site systems audits 15
Data evaluation 15
Quality assurance samples 16
Method of estimating analytical precision 16
Precision results for rock fragment determination 17
Precision results for bulk density determination 18
Completeness results

CONTENTS (continued)

Page

5. Conclusions and Recommendations 19
General recommendations 19
Sample receipt 19
Equipment Inventory 20
Sample drying 20
Moisture determination 20
Soil homogenization 21
Rock fragment determination 21
Qualitative test for inorganic carbon 21
Bulk density determination 21
Sample shipment 22
Quality assurance and quality control samples 22
Record keeping 22
Design components 23

References 28

Appendix A. Preparation Laboratory Manual for the Direct/Delayed Response Project 29

FIGURES

Number		Page
1	Form 101 - Master data form	2
2	Form 102 - Shipping form	4
3	Sample receipt form	24
4	Bulk density raw data form	25
5	Bulk sample processing form	26

TABLES

Number		Page
1	Precision estimates for rock fragments	16
2	Precision estimates for bulk density	17

ACKNOWLEDGMENTS

Critical peer reviews by the following individuals are gratefully acknowledged: W. Banwart, University of Illinois, Urbana, Illinois; and J. S. Lohse, Illinois Department of Agriculture, Bureau of Farmland Protection, Springfield, Illinois.

The following individuals provided guidance in the development of statistical analyses: T. Starks, Environmental Research Center, University of Nevada, Las Vegas, Nevada; and J. E. Teberg and M. J. Miah, Lockheed Engineering and Management Services Company, Inc., Las Vegas, Nevada.

Critical reviews by the following individuals were instrumental in the preparation of this document and are gratefully acknowledged: C. J. Palmer, Environmental Research Center, University of Nevaća, Las Vegas, Nevada; J. C. Foss, University of Tennessee, Knoxville, Tennessee; B. R. Smith, Clemson University, Clemson, South Carolina; J. J. Lee, EPA Environmental Research Laboratory, Corvallis, Oregon; D. S. Coffey, Northrop Services, Inc., Corvallis, Oregon; and J. K. Bartz, M. D. Best, G. E. Byers, W. H. Cole, and M. L. Papp, Lockheed Engineering and Management Services Company, Inc., Las Vegas, Nevada.

SECTION 1

INTRODUCTION

OVERVIEW

The U.S. Environmental Protection Agency (EPA), in conjunction with the National Acid Precipitation Assessment Program (NAPAP), has designed and implemented a research program to predict the long-term response of watersheds and surface waters in the United States to acidic deposition. Based on this research, each watershed system studied will be classified according to the time scale in which it will reach an acidic steady state, assuming current levels of acidic deposition. The Direct/Delayed Response Project (DDRP) was designed as the terrestrial complement to the aquatic resources program.

As part of the DDRP, the services of two preparation laboratories were obtained through interagency agreements to receive and process soil samples collected from the Southern Blue Ridge Province (SBRP) of the United States and to perform preliminary analyses on these samples. Laboratories located at the University of Tennessee in Knoxville, Tennessee and at Clemson University in Clemson, South Carolina were selected for these tasks because of the proximity of each laboratory to the sampling sites and analytical experience with soils of the region.

Each laboratory was supervised by a university faculty member and a laboratory manager. The manager was responsible for ensuring that the integrity of the soil samples was maintained after the samples were delivered to the preparation laboratory. Both laboratory managers had received university degrees in soil science and the other laboratory personnel had received prior training in soil science. All participants were required to comply with specified protocols, as outlined in Appendix A.

Soils processed at the preparation laboratories were collected from sampling sites located in Georgia, South Carolina, North Carolina, and Tennessee and from special interest watersheds in North Carolina and Virginia. Upon receipt of bulk soil samples and clod samples from the sampling crews, laboratory personnel performed the following analyses on the samples:

- Moisture determination (air-dry moisture content)
- Rock fragment determination (2- to 4.75-millimeter and 4.75- to 20-millimeter fractions by weight)
- Qualitative test for inorganic carbon
- Bulk density determination by clod analysis

Laboratory personnel prepared analytical samples derived from homogenized, air-dry bulk samples. The analytical samples were labeled and were organized according to their parent pedons. Multiple batches were assembled, each containing no more than 37 routine and duplicate samples, two natural audit pairs, and a preparation duplicate. The duplicates and audit pairs were included in each batch for quality assurance (QA) purposes. The samples were randomized within each batch by the laboratory manager. The assembled batches were shipped to various analytical laboratories contracted by EPA for additional analyses.

The preparation laboratories were responsible for completing Form 101 (see Figure 1) for each batch of samples. This data form contained the results of all analyses performed on the soil samples by

NATIONAL ACID DEPOSITION SOIL SURVEY (NADSS)
FORM 101

DATE RECEIVED BY DATA MGT. D D M M Y Y

Batch ID _____
Crew ID _____
Prep Lab ID _____
Lab Set Sent To _____
Date Shipped _____

Set ID	_ _ _ _	_ _ _ _	_ _ _ _
Date Sampled	_ _ _ _ _ _ _ _	_ _ _ _ _ _ _ _	_ _ _ _ _ _ _ _
Date Received	_ _ _ _ _ _ _ _	_ _ _ _ _ _ _ _	_ _ _ _ _ _ _ _
Date Prep Completed	_ _ _ _ _ _ _ _	_ _ _ _ _ _ _ _	_ _ _ _ _ _ _ _
No. of Samples	_ _		

SAMPLE NO.	SITE ID	SAMPLE CODE	SET ID	COARSE FRAGMENTS % CF	AIR DRIED MOISTURE % W	AIR DRIED MOISTURE % RSD	INORG. CARBON (IC) Y = YES N = NO	BULK DENSITY g/cc
	******	**********	****	**.**	**.**	**.**		**.**
01								
02								
03								
04								
05								
06								
07								
08								
09								
10								
11								
12								
13								
14								
15								
16								
17								
18								
19								
20								
21								
22								
23								
24								
25								
26								
27								
28								
29								
30								
31								
32								
33								
34								
35								
36								
37								
38								
39								
40								
41								
42								

Signature of Preparation Laboratory Manager: _____
Comments:

WHITE – ORNL CANARY – PREPARATION LAB PINK – ENSL LY GOLD – ERL C GILLS (782) 352-2-00

Figure 1. Form 101 - Master data form

preparation laboratories prepared a shipping form, Form 102 (see Figure 2), that was included with each batch of samples shipped to the analytical laboratories. The shipping form identified each sample by a batch and sample number accompanied by a mineral or organic soil designation. This procedure disguised the originating pedon and horizon of each field sample, as well as the identity of the QA samples.

All information, e.g., sample labels, received from the sampling crews and raw data from the preparation laboratory analyses were documented in log books. The protocols specified that completed log books were to be sent to EMSL-LV QA staff for use during the data verification process.

QA and quality control (QC) measures were applied to maintain consistency in soil preparation protocols and to ensure that the soil sample analyses would yield results of known quality. Laboratory personnel received training in the preparation procedures and analytical methods. QA representatives from EMSL-LV and ERL-C conducted on-site systems audits of the preparation laboratories. Weekly communication between the QA staff and laboratory personnel was established to identify, discuss, and resolve issues.

OBJECTIVES

This document reports the results of the preparation laboratory operations and QA program for the SBRP Soil Survey. Information concerning the specified protocols for the preparation laboratories can be found in Appendix A.

The following sections contain detailed information concerning the preparation laboratory methods, operations, and data quality. A series of recommendations for improving both the quality and efficiency of preparation laboratories in future DDRP soil surveys are included.

NATIONAL ACID DEPOSITION SOIL SURVEY (NADSS)
SHIPPING FORM 102

DATE RECEIVED
BY DATA MGT. D D M M M M Y Y

Prep Lab ID _____ Date Received D D M M M Y Y

Batch ID _____ Date Shipped _ _ _ _ _ _ _

Analytical Lab ID _____

SAMPLE NO.	AIR-DRIED MOISTURE % W	AIR-DRIED MOISTURE % RSD	INORGANIC CARBON (IC) Y = YES N = NO	COARSE FRAGMENTS SHIPPED? (CHECK Y IF YES)
	- - . - - -	- - . - -	-	
01				
02				
03				
04				
05				
06				
07				
08				
09				
10				
11				
12				
13				
14				
15				
16				
17				
18				
19				
20				
21				
22				
23				
24				
25				
26				
27				
28				
29				
30				
31				
32				
33				
34				
35				
36				
37				
38				
39				
40				
41				
42				

Signature of Preparation Laboratory Manager: _____

Comments:

WHITE – SMO CANARY – ANALYTICAL WITH COPY TO SMO PINK – ANALYTICAL WITH COPY TO EMSL-LV GOLD – ANALYTICAL LAB

GILLS (702) 362-2100

Figure 2. Form 102 - Shipping form

SECTION 2

SAMPLE PREPARATION METHODS AND ANALYSIS

The detailed methods and analytical procedures used in soil preparation activities for the SBRP soil survey are given in the protocols (Appendix A). Brief explanations of the specified methods and procedures for the preparation laboratory tasks are given below, including one correction to the protocols. The procedures are discussed in sequential order of performance by the preparation laboratories.

SAMPLE DRYING

Bulk samples are spread out on large sheets of paper to air dry. Where necessary, several sheets of paper are placed underneath the samples to absorb excess moisture and the paper is replaced periodically for faster drying. Laboratory personnel occasionally stir the samples to encourage uniform drying.

MOISTURE DETERMINATION

A 15- to 20-gram air-dry subsample is placed in a drying dish and is weighed. The dish is placed in a drying oven for a minimum of 16 hours, 105°C for mineral samples and 60°C for organic samples. The sample is allowed to cool in a desiccator for 30 minutes and is reweighed. This procedure is repeated until the difference between successive daily moisture contents is less than 2.5 percent. The final air-dry moisture content is recorded. (Note: Field moisture was not a measured parameter for the SBRP survey.)

CRUSHING AND SIEVING

After recording the weight of the air-dry bulk sample, the soil peds are crushed to allow passage of the less than 2-mm soil fraction through a No. 10 sieve. A wooden rolling pin or a rubber stopper is used to crush the peds, depending on the consistence of rock fragments in each sample.

ROCK FRAGMENT DETERMINATION

Rock fragments remaining from the sieving procedure are retained on either one of two sieves. Fragments retained on the No. 4 sieve constitute the 4.75- to 20-millimeter coarse pebble fraction (fragments larger than 20 millimeters in diameter were sieved by the sampling crews). Fragments retained on the No. 10 sieve constitute the 2.0- to 4.75-millimeter fine pebble fraction. Gravimetric analysis is used to determine the percentage by weight of each fraction in the soil samples.

SOIL HOMOGENIZATION

A Jones-type riffle splitter is used for homogenizing the samples. The less than 2-millimeter fraction is deposited evenly across the baffles of the riffle splitter and is channeled into two receiving pans. After seven passes through the riffle splitter, the entire sample is combined for a final pass through the riffle splitter. The material in one pan is placed into a plastic sample bag for archiving, and

the material in the other pan repeatedly passes through the riffle splitter until a one-kilogram subsample is obtained.

QUALITATIVE TEST FOR INORGANIC CARBON

One gram of air-dry soil is placed in the well of a porcelain spot plate, is saturated with deionized water, and is stirred to release any entrapped air. The sample is observed through a microscope or a stereoscope in order to detect an effervescent reaction when three drops of 4N HCl are added. Two types of QC samples are used to determine the detection limit and to qualitatively calibrate the test.

BULK DENSITY DETERMINATION

Replicate soil clods, usually three per horizon, are collected by the sampling crews (Blume et al., 1987). Initially, the clods are weighed at the laboratory and are dipped in a 1:7 saran:acetone mixture. The clods are suspended from a line, are allowed to dry briefly, and are reweighed. The dipping procedure is repeated until each clod is assumed to be impervious to water.

Approximately 800 milliliters of deionized water in a one-liter beaker is de-gassed by boiling, is allowed to cool to room temperature, and is tared on a balance. Each clod is submerged in the water and the increase in weight is recorded. The clods are oven-dried for 48 hours, cooled, and re-weighed. A two-hour heat treatment in a $400^{o}C$ muffle furnace allows the saran to vaporize, and the clods are cooled and again are reweighed. Each clod is crushed and is passed through a 2-millimeter sieve to determine percent by weight of rock fragments. This figure is used to adjust the bulk density calculation for rock fragment content.

Bulk density is defined as the mass of dry soil per unit volume, including pore space, and expressed as grams per cubic centimeter (g/cm^3). Bulk density normally ranges from 1.0 to 1.8 g/cm^3 in mineral soils (USDA-SCS, 1983).

Laboratory personnel at the Clemson preparation laboratory discovered that one of the bulk density algorithms was given incorrectly in the protocols. The algorithm used to estimate the air-dry saran weight was originally written as:

$$M_{TS} = \frac{X\,(M_a - M_1)}{a - 1}$$

where: M_{TS} = air-dry saran weight

X = total number of coatings (field and laboratory)

M_a = clod weight after final coating

M_1 = initial clod weight after unpacking

a = number of laboratory coatings

The correct algorithm is as follows:

$$M_{TS} = \frac{X\,(M_a - M_1)}{a}$$

All participants were informed of the error as soon as it was discovered. The laboratory data forms immediately were amended to show the corrected values.

SECTION 3

PREPARATION LABORATORY OPERATIONS

This section describes preparation laboratory operations, including difficulties with the procedures and deviations from the protocols. The laboratories are characterized as Laboratory A and Laboratory B to prevent disclosure of their identity, except where necessary to clarify an activity.

SAMPLE RECEIPT AND STORAGE

Due to budgeting and logistical difficulties, neither preparation laboratory was administratively operational when sampling began. The laboratory director at Laboratory B retired from the position soon after preparation activities were initiated, causing some initial confusion with sample tracking and processing.

Each laboratory provided the sampling crews with convenient access to cold storage. A sample receipt log book was kept at each facility to allow sampling crews to log in the samples. Each laboratory was responsible for checking that all samples delivered by the sampling crews were recorded in the log book. Entries were checked for legibility and accuracy.

The temperature of the storage facilities was maintained at the contract-specified $4^{o}C$, with one exception. Laboratory B had a temporary refrigeration failure that was reported to the QA staff immediately. The refrigeration unit was repaired within approximately six hours, and the highest temperature measured during the failure was $14^{o}C$. The lapse in cooling may have had some effect on microbial activity in field-moist samples stored at the facility, although studies were not conducted to identify this effect. Analytical data for the affected samples processed at Laboratory B will be flagged in the verified data base, denoting possible contamination. If time permits, the data will be evaluated during verification to determine whether or not there are suspect values that indicate contamination may have occurred. Although the effects are expected to be negligible, organic transformations involving carbon, nitrogen, or sulfur may have occurred. This issue will be discussed in the QA report for the analytical data.

In order to provide an equal distribution of incoming samples and to improve the efficiency of available cold storage and labor, 34 bulk samples and 72 corresponding clods were transferred from the University of Tennessee to Clemson University. The samples were packed in coolers and were transported by truck. The samples were returned to cold storage within eight hours. This transfer provided each laboratory with approximately 400 bulk samples and their respective clods.

There was some confusion concerning the proper dispensation of practice samples that were delivered to Laboratory B at the request of the laboratory director. These practice samples had been collected by one of the sampling crews in preparation for routine sampling. The samples were delivered to Laboratory B and were logged into the sample receipt log book. The samples were not identified as practice samples, therefore laboratory personnel treated the samples as if they were routine samples and processed them accordingly. The error was discovered after the samples had undergone soil analysis at the analytical laboratories. Data for the practice samples were later removed from the data base.

EQUIPMENT INVENTORY

The laboratory managers were responsible for tracking the distribution of equipment to the sampling crews. Each preparation laboratory stored the equipment in a locked room with limited access. The sampling crews usually obtained supplies while samples were being delivered to the cold storage facility. Laboratory managers generally were notified in advance of a sampling crew's arrival to ensure that the crews had access to the supply room. The sampling crews were asked to record in the equipment log book all supplies taken.

A complete inventory was performed monthly at each preparation laboratory. Equipment shortages were reported to EMSL-LV during the weekly conference call. Laboratory personnel were tasked with providing the saran and acetone used in mixing a solution for coating clods in the field. They also were responsible for providing gel packs used in the refrigeration of bulk samples during transport.

It was decided at the training workshop that certain sampling hardware would be distributed to the sampling crews via the Soil Conservation Service (SCS) state offices instead of the preparation laboratories. After sampling was completed, difficulties were encountered in tracking and recovering this equipment because the laboratory managers were not provided with a master inventory sheet listing all equipment provided to the crews. Additional difficulties occurred when sampling crews utilized both preparation laboratories as supply points.

After the soil preparation activities were completed, leftover supplies were inventoried and were sent to EMSL-LV for storage. Cameras used to photograph the sampling sites were turned over to the SCS state offices by some of the sampling crews. Although there were instances of misplaced sampling hardware, most of the equipment eventually was located. A complete list of the types of equipment supplied to the crews is provided in Appendix A.

SAMPLE DRYING

Although there were no deviations from the specified protocols for sample drying, concerns were raised about air-drying the soils during humid or rainy weather. Drying areas for both laboratories were located in buildings lacking humidity control: one used a greenhouse and the other used a prefabricated metal structure. Samples occasionally took as long as three weeks to dry. Long-term exposure of the samples during humid weather provided a greater opportunity for contamination. Both laboratories loosely covered the drying samples with kraft paper to reduce airborne contamination.

MOISTURE DETERMINATION

One significant deviation from the specified protocols for the air-dry moisture determination occurred. Laboratory B obtained a convection oven after the sample processing had begun. Therefore, the initial air-dry moisture determinations at this laboratory were made by an alternate method approved in advance by QA staff. The alternate method was to weigh a known quantity of sample and allow it to dry overnight under the same conditions as the bulk sample. The sample then was reweighed. If the change in sample weight was less than 2.5 percent absolute over a two-day period, it was presumed that the soil was ready for processing. If not, the procedure was repeated until the variation in weight was less than 2.5 percent.

CRUSHING AND SIEVING

There were no deviations from the specified protocols for sample crushing and sieving. Laboratory A generally used the rolling pin method. Because of the soft, weathered state of rock fragments in its samples, Laboratory B generally used the specified alternate method, which utilized a rubber stopper to crush individual soil peds. This procedure allowed the passage of soil through the sieves without disturbing the fragments. The rolling pin method occasionally was used by Laboratory B for those samples containing hardened peds or few fragments, and a notation of this was made in the sample processing log book.

ROCK FRAGMENT DETERMINATION

There were no deviations from the specified protocols for rock fragment determination. Laboratory B washed all rock fragments in deionized water to remove adhered soil before calculating the percentages. Both laboratories bagged and labeled the fragments and awaited further instructions for shipment.

SOIL HOMOGENIZATION

There were no deviations from the specified protocols for soil homogenization. During October, 1986 it was discovered that there was insufficient audit material remaining to continue processing one-kilogram audit samples. As a result, the protocols were modified and the laboratory managers were advised to begin preparing 500-gram analytical samples instead of one-kilogram samples. All batches shipped from Laboratory A contained 500-gram samples. Because many of the batches from Laboratory B already had been shipped, only 5 of the 14 total batches at Laboratory B contained 500-gram samples.

Both laboratory managers voiced concern about rehomogenizing the bulk samples to obtain subsamples for mineralogical study. Repetitious soil homogenization could misrepresent the particle-size distribution because of the potential for loss of clay and fine silt particles during riffle-splitting. The list of samples selected for mineralogical analysis was sent to the preparation laboratories after soil processing was well underway. Bulk samples had to be retrieved from storage, rehomogenized, subsampled, relabeled, and returned to storage. This exercise was very time consuming and might have been avoided through better planning and communication.

QUALITATIVE TEST FOR INORGANIC CARBON

There were no deviations from the specified protocols for the inorganic carbon test. Inorganic carbon was not detected in any samples collected.

BULK DENSITY DETERMINATION

There were no deviations from the specified protocols for the bulk density determination. Clod analysis was chosen as the method for determining bulk density, despite a few disadvantages. Obtaining replicate clods from dry, loose, or extremely wet soils and from horizons containing many rock fragments often can be difficult or impossible. Another concern is that the bulk density values that are obtained may be higher than the average bulk density of the soil horizon they represent, because

sampling could be biased toward the collection of firmer, more coherent clods capable of withstanding disturbance during sampling and transport.

Variations in bulk density measurements could occur when clods are allowed to air dry after multiple saran coatings. Because the saran is permeable to water vapor, the clod weight could be affected by moisture loss from prolonged drying. This situation did occur at Laboratory A during the early stages of sample processing, although the actual number of affected clods is unknown. Thirty-nine clods at Laboratory B were discarded because a weak coating solution used in the field rendered the clods unsuitable for analysis upon arrival at the laboratory.

According to the log books, three floating clods were identified. Each clod was forcibly submerged to allow a bulk density determination to be made.

SAMPLE SHIPMENT

The preparation laboratories were provided with shipping materials used for packaging the samples and with a express mail charge number for overnight shipment of the samples to the analytical laboratories. Laboratory personnel were instructed to assemble batches containing as many as 37 routine samples and field duplicates. Each batch also contained one preparation duplicate prepared by the laboratory and two pairs of natural audit samples provided by QA staff at EMSL-LV. The laboratory managers were responsible for randomizing the samples within each batch and assigning sample numbers. The individual samples were tagged with the appropriate label B and were packed in cardboard boxes for shipment to the designated analytical laboratory.

QA representatives routinely called one or two days in advance to notify the preparation laboratory manager that a shipment was due for delivery to an analytical laboratory. Laboratory B expressed some dissatisfaction with the amount of advance notice received. In a few cases, notification was given the day before batches were to be shipped. Laboratory A was not directly affected because their batches generally were sent in two mass shipments.

Because of insufficient detail in the protocols, there was some confusion in the handling of the audit samples. Later written instructions stated that the audit samples should be identical in appearance to the routine samples, although the audit samples had arrived at the preparation laboratories with numbers on both the outer canvas bags and inner plastic bags. The integrity and anonymity of the audit samples were jeopardized because Laboratory A left the audit samples in their original bags and Laboratory B re-bagged the audit samples.

RECORD KEEPING

Both preparation laboratories were provided with log books to use for recording data. Each laboratory manager was instructed to organize log books containing the following information:

- Label A - This log book contained labels that originally were filled out by the sampling crews and were affixed to the inner bag of the bulk samples. Laboratory B placed these labels on loose-leaf paper and organized them into a binder. Laboratory A did not submit their labels, and later efforts to locate them were unsuccessful.

- Clod Label - This log book contained labels that were filled out by the sampling crews and affixed to the prepared clods. Laboratory A did not submit their labels, and later efforts to locate them were unsuccessful.

- Sample Receipt - This log book was filled out by the sampling crews upon delivery of samples to each laboratory.

- Equipment - The sampling crews were asked to list the field supplies they obtained at the laboratories. The laboratory managers were responsible for tracking and confirming the equipment inventories.

- Percent Moisture - This log book contained raw data from the air-dry moisture analyses.

- Percent Rock Fragments - This log book contained raw data from the rock fragment analyses.

- Bulk Density - This log book contained raw data from the bulk density analyses.

- Inorganic Carbon - This log book contained raw data from the test for inorganic carbon.

- Sample Processing - This log book tracked the progress of the soil samples through the various preparation activities.

Because a standard format for each log book was not specified, there was variation between the laboratories. As a result, verification of the data took more time than was expected. Laboratory A assigned internal laboratory numbers to their samples, making it difficult for QA personnel to match these numbers to the original sample codes.

SECTION 4

QUALITY ASSURANCE/QUALITY CONTROL

A specified QA/QC program must be followed during the course of survey activities to ensure that the resulting data are of known quality. The QA/QC program for the SBRP soil survey consisted of design and evaluative components that aided survey participants in obtaining samples and producing data that meet the needs of end users.

The QA/QC design for the preparation laboratories included training personnel in the protocols to be followed, establishing a communications network, assessing data quality, and performing on-site systems audits. The data were evaluated systematically using analytical data from the clod replicates and from the QA duplicate samples that were included in each batch of routine samples. The following sections explain aspects of the QA/QC program in relation to preparation laboratory activities.

DESIGN COMPONENTS

Training

Preparation laboratory personnel attended a regional workshop held in Knoxville, Tennessee, from March 18 through 20, 1986. The purpose of the workshop was to review the laboratory protocols and discuss key activities. Training of the participants established a basis for consistency between the laboratories, thereby increasing the likelihood that the data would be of comparable quality.

Communications

Weekly conference calls assisted in keeping the preparation laboratories operating efficiently and consistently by providing a forum that allowed potential difficulties to be identified, discussed, and resolved. Preparation laboratory managers, QA personnel, and scientists involved in the study of soil mineralogy participated in these calls. Issues discussed during the conference calls included supply shortages and clarification of procedures, e.g., sample labeling, record keeping, and drying of clods. The laboratory manager at Laboratory A stated that the conference calls were not as beneficial as routine calls for dealing with specific issues.

Data Quality Objectives

Data quality objectives for the preparation laboratories were not established during the SBRP soil survey. The preparation laboratories were assessed according to the following data characteristics:

- Precision and accuracy - These are quantitative measurements that estimate the amount of variability and bias inherent in a given data set. Precision refers to the level of agreement among repeated measurements of the same parameter. Accuracy refers to the difference between an estimate based on the analytical results and the true value of the parameter being measured.

- Representativeness - This refers to the degree to which the collected data accurately reflect the population or medium that is sampled.

- Completeness - This refers to the amount of data that is successfully collected with respect to the amount intended in the design. A defined percentage of the intended amount must be successfully collected for conclusions based on the data to be valid. Lack of data completeness

may reduce the precision of estimates, may introduce bias, and may reduce the level of confidence in the conclusions.

- o Comparability - This refers to the similarity of data from different sources included in a single data set. If more than one laboratory is analyzing samples, uniform procedures must be used to ensure that the data from different sources are based on measurements of the same parameter.

These five data quality characteristics were identified in the DDRP QA Plan, and their application to preparation laboratory activities were stated (Bartz et al., 1987). A brief description of the specific characteristics follows:

- o Precision and Accuracy - The preparation laboratory combines sets of field samples into one batch containing a maximum of 39 routine and duplicate samples. After processing, i.e., air-drying, crushing, sieving, and homogenization, one bulk sample is split into two subsamples which are termed preparation duplicates. Comparison of physical and chemical data for these duplicates allows evaluation of the subsampling procedure.

- o Representativeness - Each bulk sample is processed by a preparation laboratory to obtain a homogeneous sample. Homogenization is accomplished by passing the sample through a Jones-type riffle splitter at least seven times. The riffle splitter also is used for subsampling. All samples not being processed are stored at $4^{o}C$ by the preparation laboratory.

- o Completeness - Each batch of samples sent to a contractor analytical laboratory includes the preparation duplicates.

- o Comparability - All preparation laboratories process bulk samples according to the protocols. Strict adherence to protocols should result in comparability among preparation laboratories.

Precision is estimated in this QA report by evaluating field duplicate data from the rock fragment analyses and replicate data from the bulk density analyses. Additional precision estimates using analytical data from the field duplicates and from the preparation duplicates will be documented in the QA report on the analytical laboratory data.

Accuracy cannot be assessed because: (1) true values for the parameters in question are not known, (2) quality control calibration samples were not used in the determination of bulk density, (3) it was not known how to provide quality control calibration samples to evaluate the rock fragment analyses, and (4) it was not known how to provide audit samples for the rock fragment and bulk density analyses. Relative accuracy based upon an interlaboratory study will be addressed in the QA report on the analytical laboratory data.

Representativeness will be addressed in the QA report on the analytical data by using data from the preparation duplicates to assess the subsampling procedure performed by each laboratory.

Completeness is measured by each laboratory's performance of the analyses and processing tasks for all samples assigned.

Comparability is evaluated using the precision data from preparation duplicates. This evaluation will be documented in the QA report on the analytical laboratory data.

On-Site Systems Audits

Pre-sample audits of both preparation laboratories were conducted during the Spring of 1986. These visits were designed to assess the laboratory facilities, equipment, and staff before any samples were received or processed. Laboratory A was audited by a QA representative from EMSL-LV, and Laboratory B was audited by a QA representative from ERL-C.

Laboratories A and B later were audited by a QA representative from EMSL-LV in June, 1986. At Laboratory A, the QA auditor observed sample drying and toured the cold storage and laboratory facilities. At Laboratory B, the auditor observed the early stages of sample preparation, including the sample drying, sample crushing, and soil homogenization procedures.

Laboratory A had not been covering their drying samples with paper, thereby leaving the samples vulnerable to airborne contamination. The laboratory manager was instructed to loosely cover all samples in the future. Laboratory B was not calibrating the thermometer used for recording the temperature of the water in bulk density calculations. The laboratory manager was instructed to check the accuracy of the thermometer at least once a month. It also was discovered that Laboratories A and B did not have the required Class S weights for calibration of the balance. These weights were obtained shortly thereafter.

The log books were inspected by the QA auditor, who observed pencil entries in the sample receipt log book at Laboratory B. To correct this deficiency, a black pen was attached to the log book and was used thereafter by the sampling crews. Laboratory A occasionally used blue ink instead of the specified black ink. Although both laboratories set up their log books with different formats and headings, the books contained all required data.

DATA EVALUATION

Quality Assurance Samples

Three types of QA samples were included in each batch of samples submitted to the analytical laboratory: (1) field duplicates, (2) preparation duplicates, and (3) natural audit samples. Data from the QA samples will be evaluated in the QA report for the analytical laboratory data. An explanation of each type of QA sample follows.

One horizon per sampling crew per day was sampled in duplicate as specified in the field protocols (Blume et al., 1987). The first sample of the pair is considered the routine sample, and the second sample is referred to as the field duplicate. The field duplicate underwent the same preparation steps as its associated routine sample. This allows an estimate to be made of the combined effects of sampling error, horizon variability, and preparation laboratory error.

One sample per batch was chosen by the laboratory manager to be processed and then split into two subsamples. On the Form 101 sent to EMSL-LV, one of the pair retained the routine sample code and the other was assigned the preparation duplicate designation. Analytical data from the preparation duplicates allow the range of physical and chemical characteristics for splits of the sample material to be determined. Statistical analyses of the data will allow an estimate to be made of the within-laboratory variability due to subsampling.

Two natural audit pairs supplied by QA staff were included in each batch that was sent from a preparation laboratory to an analytical laboratory, but the samples did not undergo any processing at

the preparation laboratory. These samples were used to assess the performance of the analytical laboratories. Specific information on audit samples can be found in Bartz et al. (1987).

Method of Estimating Analytical Precision

Data for the rock fragment and bulk density determinations were grouped by laboratory into data sets. A scatter plot of horizon standard deviation versus horizon mean was generated to evaluate the relationship between precision and concentration. The data for both parameters displayed a random pattern of standard deviation, indicating that precision was independent of concentration. On this basis, a completely randomized design model was selected for the statistical estimation of precision (Steel and Torrie, 1960). The model that represents data collected at a specific sampling site can be demonstrated, as follows:

$$y_{ij} = u + h_i + e_{ij}$$

where: y_{ij} = the variable of interest for the jth observation from the ith horizon represented

u = the general mean of the population

h_i = the effect of the ith horizon on the variable of interest

e_{ij} = the random error of analytical measurement for the jth observation from the ith horizon represented

The model was used to perform statistical analyses of (1) rock fragment data from the field duplicates and (2) bulk density data from the replicate clods. A root mean square error statistic was used to estimate the pooled standard deviation (Sp) across all pedons and horizons for each laboratory data set, and the coefficient of variation (CV) was derived by dividing Sp by the mean of the data set (x) and multiplying by 100.

Precision Results for Rock Fragment Determination

Field duplicate data for the rock fragment determination were analyzed by the completely randomized design model to provide overall Sp and CV values for each preparation laboratory. Summary statistics incorporating these values are provided in Table 1.

TABLE 1. PRECISION ESTIMATES FOR ROCK FRAGMENTS

Laboratory	Field Duplicates Number of Pairs	Rock Fragments Mean (percent)	Sp	CV
A	43	7.1	1.371	19.33
B	61	13.6	1.673	12.32

Because of the simplicity of the method used for determining the percentage of rock fragments in the bulk soil samples, a greater amount of the imprecision can be attributed to spatial horizon variability or sampling bias and a lesser amount to preparation laboratory bias. The field sampling imprecision may be an indication of within-horizon rock fragment variability or improper field duplicate sampling technique.

Precision Results for Bulk Density Determination

Data from the bulk density determination were analyzed by the completely randomized design model to provide overall Sp and CV values for the sets of replicate clods at each preparation laboratory. In addition, CV values were generated for two data groups representing the sets of clods which exhibited a mean bulk density that was either greater than or less than the mean bulk density of each laboratory's clods. Summary statistics for the Sp and CV values are given in Table 2.

TABLE 2. PRECISION ESTIMATES FOR BULK DENSITY

Laboratory	Number of Horizons	Bulk Density Mean (g/cm^3)	Sp	CV	CV (<x)	CV (>x)
A	262	1.28	0.159	12.47	21.09	7.77
B	267	1.27	0.118	9.30	12.82	7.18

The CV values less than and greater than the mean were evaluated in order to determine whether or not the statistical relationship of higher CV values at lower concentrations, in this case at lower bulk densities, would hold true for bulk density data. The CV values presented in Table 2 appear to confirm this relationship.

The Sp values suggest a consistency of bulk density values within a given horizon. Audit reports indicated that the sampling crews were able to choose representative clods from each horizon and that the laboratories were consistent in their use of measurement techniques. However, the exact percentage of error contributed to the Sp values by horizon variability, sampling bias, or laboratory imprecision cannot be determined because (1) inherent spatial variability made it impossible to sample identical field clods or to provide an audit sample for measurement of potential sampling bias, and (2) the preparation laboratories were not provided with audit samples to allow estimation of laboratory bias.

The following types of sampling errors could contribute to sampling bias for the bulk density replicates:

- o Collection of replicates from transitional zones or adjacent horizons
- o Mislabeling of clods
- o Inconsistent saran coating procedure
- o Variability relating to the coherency of clods

Based on the field experience of the SCS sampling crews and the fact that the various field audit reports did not indicate major deviations from the protocols, sampling bias is not presumed to have been a significant factor affecting the Sp values. Some variability in the use of saran was mentioned in a few audit reports, although the Sp values would not be affected if the coating procedure was consistent for all replicates within a horizon.

Bias may be introduced at the preparation laboratory because of measurement or method errors, such as the following:

- o Transcription errors, such as misrecorded weights or sample codes
- o Inconsistent saran coating procedures
- o Improper clod handling, e.g., compaction
- o Incomplete drying
- o Loss of material during sieving
- o Incorrect numeric calculations
- o Faulty weights, e.g., clod tags and hairnets not subtracted, or balance not calibrated

Because audit samples were not provided, interlaboratory bias could not be estimated. Therefore, it is difficult to quantify the potential effect of preparation laboratory bias on the Sp and CV values.

Completeness Results

The requested analyses and soil processing steps were performed on 100 percent of the bulk samples and clods received by the preparation laboratories. This satisfied the maximum theoretical level of completeness. Preparation duplicates were created for each batch of samples sent to the analytical laboratories for a 100 percent level of completeness.

SECTION 5

CONCLUSIONS AND RECOMMENDATIONS

The conclusions and recommendations discussed below have been summarized from information supplied by preparation laboratory personnel, QA staff, and other survey participants. The recommendations are presented for consideration and possible implementation in future surveys. Many of the recommendations are amendments to the existing protocols and are based on the information provided in Section 3.

GENERAL RECOMMENDATIONS

In future surveys, the preparation laboratories should be on-line and operational before field sampling begins. This would allow an opportunity to assess the overall preparation function of the laboratories and to clarify the protocols in advance of soil processing activities.

The protocols should be rewritten to include an opening section that addresses general laboratory procedures. The following practices will help to improve laboratory safety and ensure the integrity of the soil samples:

- o It should be specified that eating, drinking, and smoking is prohibited in the soil drying and processing areas.
- o Inexpensive rubber or plastic gloves should be worn during all bulk sample processing activities.
- o An air-filtering mask or respirator should be worn while homogenizing soils in order to prevent excessive inhalation of airborne soil particles.
- o Samples should be crushed, sieved, and subsampled under an exhaust hood to limit airborne contamination.
- o The saran and acetone should be mixed only under an operable fume hood. A respirator also should be worn.

SAMPLE RECEIPT

Preparation laboratory personnel should be available to receive samples from the sampling crews and to check the sample labels against the log book entries. If this is not feasible, the check should be performed the following day. All discrepancies should be communicated to the QA manager within three days. A computer spreadsheet should be kept current, showing the status of all samples delivered to the laboratories.

The delivery of practice samples to a preparation laboratory is not encouraged. If such samples are delivered, the laboratory manager should ensure that the practice samples are separated from the routine samples. Under no circumstances should practice samples be logged into the sample receipt log book.

EQUIPMENT INVENTORY

The preparation laboratories should be the sole distribution and recovery points for all sampling equipment provided by EMSL-LV. A master inventory of equipment should be kept current and should be inspected by the QA auditor during the systems audits. The whereabouts of missing equipment should be determined as quickly as possible.

SAMPLE DRYING

Implementation of the following modifications to the sample drying procedure could expedite drying considerably:

- o Stirring the samples every 24 hours would encourage rapid drying.
- o In addition to the paper placed underneath the samples, a single sheet should be placed loosely over the samples. The paper beneath the samples should be replaced daily.
- o Small, clip-on, multi-speed electric fans can be used to facilitate air movement in the drying area. The fans should not be allowed to blow directly on the samples.

MOISTURE DETERMINATION

Many participants stated that a field moisture determination would be very helpful for correlating the physical and chemical status of the soil at the time of sampling. Three different methods for making this determination have been identified, as follows:

(1) Field moisture of clods: This method would utilize the same clods sampled for bulk density determination. To prevent evaporation, clods would require greater care in packaging and refrigeration before being analyzed in the laboratory. A weight measurement would be made for each clod upon arrival at the laboratory, and would be used to generate percent field moisture values upon completion of the bulk density analyses. Sampling crews would identify saturated soil horizons on the field data forms.

(2) Field moisture of bulk samples: Laboratory personnel would select a representative 20-gram mixed subsample from each bulk sample upon its arrival at the laboratory. The subsample would be placed in a capped drying container prior to weighing and oven-drying. Sampling crews would identify saturated soil horizons on the field data forms.

(3) On-site field moisture: Sampling crews would select replicate moisture samples from each soil horizon sampled and place each sample in a capped drying container. The samples would be weighed and oven-dried at the preparation laboratory. Saturated horizons would be identified on the field data forms.

Moisture data for saturated soils would be tagged automatically in the data base, irrespective of the method used to determine field moisture. The gravitational water content of a saturated sample is expected to vary widely, depending on the soil textural class, length of storage, and representativeness of the bulk sample and subsample taken.

Review of the air-drying procedure has generated the following recommendation:

- o When the laboratory manager believes that a soil sample is air dry, 10-gram subsamples should be analyzed for percent moisture on two successive days.

SOIL HOMOGENIZATION

Both laboratory managers expressed displeasure with the amount of time that elapsed before a list of the soils chosen for mineralogical study was provided. Every effort should be made to ensure that this list is provided to the preparation laboratories before the analytical samples are split from the bulk samples. This would eliminate the need for laboratories to locate and homogenize the samples twice, thereby saving time and maintaining sample integrity.

ROCK FRAGMENT DETERMINATION

Dispensation of the rock fragments following analysis should be decided before soil processing begins. This action will avoid lengthy or costly storage of the fragments at the preparation laboratories.

QUALITATIVE TEST FOR INORGANIC CARBON

Many participants considered the inorganic carbon test to be inadequate for identifying the soils that should undergo further analysis for carbonate. It has been suggested that a pH determination in 0.1M $CaCl_2$ on field-moist samples would be a better way of identifying those soils likely to contain inorganic carbon in measurable concentrations. This analysis also would provide valuable data that cannot be obtained after the samples have been air-dried. A pH value of 6.0 or higher is a possible pH at which to target individual samples for further analysis. If selected, this procedure should be performed as soon as the samples arrive at the preparation laboratory. The procedure outlined in Cappo et al., 1987 is recommended, except that a field moist sample would be used.

BULK DENSITY DETERMINATION

To avoid some of the difficulties encountered with the bulk density determinations, the following recommendations are made:

- o Audit samples and quality control calibration samples should be used during clod analysis (see recommendations under QA/QC samples).

- o Clods should be air dry before being analyzed for bulk density. The air-dry state can be assumed when the clod weight is constant, using criteria similar to those applied to the bulk samples.

- o Clod weights should be recorded immediately before coating with saran and immediately after the saran coating has dried in the laboratory.

- o A method for measuring the bulk density of floating clods should be added to the protocols. Floating clods should be identified on the bulk density raw data form and in the bulk density log book.

- The density of rock fragments within each clod should not always be assumed to be 2.65 g/cm^3 as suggested in the protocols. In areas with a variety of lithologies or weathering characteristics, an appropriate low or high density value should be used where necessary.

SAMPLE SHIPPING

Both laboratory managers stated that a shipping schedule would have helped to coordinate labor and space requirements for sample processing. Laboratory personnel often rushed to get batches shipped because of insufficient notification, usually one-day notice. A tentative schedule should be in place before the initiation of field sampling, and the schedule should be finalized immediately following the award of analytical laboratory contracts.

All necessary shipping materials, e.g., forms and labels, should be delivered to each preparation laboratory well in advance of the first shipment of batches to an analytical laboratory. This will allow an opportunity to review the forms and to resolve any issues relating to the shipping procedure or the sample packaging. Information about the audit samples should be transmitted by EMSL-LV at least one week before batch shipment, which will allow the laboratory manager time to assemble the batches and prepare Forms 101 and 102. The Form 101 for each batch should be sent to EMSL-LV on the same day the batch is shipped.

QUALITY ASSURANCE AND QUALITY CONTROL SAMPLES

Guidelines for the packaging of audit samples should be better defined in the protocols. EMSL-LV should demonstrate the sample packaging procedure during the pre-sample audit visit so that laboratory personnel do not have to repackage audit samples. To ensure anonymity of the samples at the analytical laboratories, preparation laboratory personnel should prepare the routine and duplicate samples to be indistinguishable from the audit samples.

Calibration samples should be utilized for QC checks during the bulk density determination. It has been suggested that minerals of known density, e.g., quartz at 2.65 g/cm^3, would be appropriate. The analyses could be monitored at regular intervals by using mineral samples that compare favorably in size and weight to the clods. While this particular technique has not been tested at EMSL-LV, calibrated mineral samples of the desired size and density are readily available from laboratory supply outlets. The use of graded sand samples also has been suggested as a QC check.

A synthetic audit sample should be utilized to estimate precision and interlaboratory bias during the bulk density determination. A set of plastic eggs that spans a wide range of displacement capabilities has been suggested as an audit medium, although prototypes of synthetic eggs have not been developed or tested.

RECORD KEEPING

Verification of data from the SBRP soil survey was difficult because the formats of the sample receipt and raw data log books varied considerably between Laboratories A and B. Formats for log books and for record keeping should be specified in the protocols and should be reiterated during the pre-sample audit visit. The assignment of internal laboratory numbers to the samples should be discouraged.

Standardized forms have been developed to record raw data from the preparation laboratories. The sample receipt form (Figure 3) would include column headings for field sample code, dates of sample collection and sample receipt, sample condition, number of replicate clods collected, and designation of field duplicates and paired pedons (Coffey et al., 1987). The bulk density raw data form (Figure 4) would include computerized column headings for field and laboratory weights, water displacement, rock fragments, and special features of the replicate clods. The bulk sample raw data form (Figure 5) would summarize data from the moisture, rock fragment, and inorganic carbon (or pH) determinations. Each bulk sample form would contain data for one sample. The completed forms could be sorted alphabetically by state, numerically by pedon number, and could be used as an index for the Form 101 data.

Laboratory personnel could enter the raw data into a data base file via a compiled data base software program, which could be accessed through a personal computer without actually using the data base software. (Note: A program similar to this was generated by ORNL and used by the University of Tennessee laboratory.) Ideally, each data entry screen would display the same format as the raw data forms. The final data could be calculated and printed on the Form 101 automatically. Entry or procedural errors could be identified before a batch is sent to an analytical laboratory. Data verification could be completed shortly after the final batch of soil samples is analyzed at the preparation laboratory. The software should be delivered and demonstrated during the pre-sample audit or the training workshop.

DESIGN COMPONENTS

It was suggested that a more extensive training workshop be conducted. The workshop should be held when all laboratory personnel are able to attend and before soil sampling has begun. Laboratory personnel could be instructed on the preparation procedures, record keeping, and packaging samples for shipment. Also, the QA auditor could demonstrate the use of the data entry computer program and could help in setting up the sample drying area.

The documentation and dissemination of the information discussed during conference calls would be improved if the following suggestions were implemented:

- The QA laboratory auditor should be the moderator for all conference calls. A staff assistant should act in the place of the QA auditor only when necessary.
- All participants in each conference call should be identified.
- Each conference should be tape recorded in order that the major points of the discussion can be clarified if necessary.
- All pertinent information should be documented by the moderator in a log book set up specifically for this purpose.
- Log book notes should be compiled monthly and should be typed and sent to all participants.

After preparation laboratory operations are underway, the conference calls may be reduced to bi-weekly or monthly intervals. The conference calls should not exclude calls of an urgent nature, because open communication and swift resolution of difficulties is in the best interest of all soil survey participants.

Sample Number	Crew ID	Site ID	Date Collected	Date Received	Time Received	Delivered By	Received By	Sample Condition	Additional Notes		
								Wet/Dry (W/D) Sieved/Unsieved (S/U) Bag Split (BS) Under Volume (UV)	Number of Clod Samples Collected for Each Horizon Sampled	Field Duplicate	Paired Pedon

Figure 3. Sample receipt form

All weights in ± 0.01 grams

BULK DENSITY RAW DATA

PREP. LAB. _____

Page __/__

SAM_CODE	REP	FIELD_OP	FIELD_WT	LAB_OP	LAB_WT	CLOD_H_2O	TEMP	CLOD_OD	R_FRAG	EXTRA	FLOAT	DATE	COMMENTS	ANALYST INITIALS

Figure 4. Bulk density raw data form

BULK SAMPLE RAW DATA

SAMPLE ID: _ _ _ _ _ _ _ _ _ _ _ _ _ _ DATE SAMPLED: ___/___/___

SITE ID: _____ DATE REC'D: ___/___/___

SET ID: _____ PROCESS START: ___/___/___

BATCH ID: _____ PROCESS COMPLETE: ___/___/___

SOIL TYPE: M / O INORGANIC CARBON: YES / NO

Initials: Initials:

SAMPLE DRYING:

Date	Weight	Initials
___/___	_____.___g	_____
___/___	_____.___g	_____
___/___	_____.___g	_____
___/___	_____.___g	_____
___/___	_____.___g	_____

TOTAL AIR DRY WT:

Date: ___/___/___

_____.___g

Initials:

ROCK FRAGMENT WT:

Date: ___/___/_____

2 to 4.75 mm: _____.___g

4.75 to 20 mm: _____.___g

Initials:

ENTERED IN COMPUTER: Date: ___/___/_____ Initials:

COMMENTS:

Figure 5. Bulk sample raw data form

Each set of systems audits should be performed by the same QA auditor, thereby ensuring uniformity in the evaluation of preparation laboratory activities. The second round of audits should be conducted after sample processing is underway. Audit reports should be submitted to the QA manager within two weeks after returning from an audit, and telephone contact should be made within two days if serious discrepancies have been identified.

REFERENCES

Bartz, J. K., S. K. Drouse, K. A. Cappo, M. L. Papp, G. A. Raab, L. J. Blume, M. A. Stapanian, F. C. Garner, and D. S. Coffey. 1987. Direct/Delayed Response Project: Quality Assurance Plan for Soil Sampling, Preparation, and Analysis. U. S. Environmental Protection Agency, Las Vegas, Nevada.

Blume, L. J., M. L. Papp, K. A. Cappo, J. K. Bartz, D. S. Coffey, and K. Thornton. 1987. Soil Sampling Manual for the Direct/Delayed Response Project Soil Survey. Appendix A In Coffey, D. S., J. J. Lee, J. K. Bartz, R. D. Van Remortel, M. L. Papp, and G. R. Holdren. 1987. Field Operations and Quality Assurance Report for Soil Sampling and Preparation in the Southern Blue Ridge Province of the United States, Volume 1: Sampling. U. S. Environmental Protection Agency, Las Vegas, Nevada.

Cappo, K. A., L. J. Blume, G. A. Raab, J. K. Bartz, and J. L. Engels. 1987. Analytical Methods Manual for the Direct/Delayed Response Project Soil Survey. EPA 600/8-87/020. U. S. Environmental Protection Agency, Las Vegas, Nevada.

Coffey, D. S., J. J. Lee, J. K. Bartz, R. D. Van Remortel, M. L. Papp, and G. R. Holdren. 1987. Field Operations and Quality Assurance Report for Soil Sampling and Preparation in the Southern Blue Ridge Province of the United States, Volume I: Sampling. U. S. Environmental Protection Agency, Las Vegas, Nevada.

Steel, R. G. D., and J. H. Torrie. 1960. Principles and Procedures of Statistics. McGraw-Hill Book Company, New York. 481 pp.

USDA-SCS. 1983. National Soils Handbook, Parts 600-606. U. S. Government Printing Office, Washington, D.C.

APPENDIX A

PREPARATION LABORATORY MANUAL FOR THE DIRECT/DELAYED RESPONSE PROJECT SOIL SURVEY

by

J. K. Bartz, D. S. Coffey, and L. J. Blume

The following protocols were used by preparation laboratory personnel during the Southern Blue Ridge Province Soil Survey. This appendix was Part III of the draft "Soil Sampling and Preparation Laboratory Manual for the Direct/Delayed Response Project Soil Survey". The draft did not undergo a full external review and was not formally released by EPA. It is presented here without editorial correction.

The protocols are preceded by a table of contents from the draft manual. Parts I and II of the manual are presented as Appendix A in Coffey et al. (1987), referenced as follows:

Coffey, D. S., J. J. Lee, J. K. Bartz, R. D. Van Remortel, M. L. Papp, and G. R. Holdren. 1987. Field Operations and Quality Assurance Report for Soil Sampling and Preparation in the Southern Blue Ridge Province of the United States, Volume I: Sampling. U. S. Environmental Protection Agency, Las Vegas, Nevada.

TABLE OF CONTENTS

Section		Page	Revision
	PART I. OVERVIEW		
1.0	INTRODUCTION	1 of 3	4
	PART II. FIELD OPERATIONS		
2.0	FIELD PERSONNEL AND EQUIPMENT.	1 of 8	4
	2.1 Personnel	1 of 8	4
	2.1.1 Field Crews.	1 of 8	4
	2.1.2 USDA Soil Conservation Service, Soils Staff.	2 of 8	4
	2.1.3 Regional Coordinator/Correlator. . .	2 of 8	4
	2.1.4 Quality Assurance/Quality Control Representative	3 of 8	4
	2.2 Field Equipment	3 of 8	4
	2.2.1 Site Selection Equipment	4 of 8	4
	2.2.2 Excavation Equipment	4 of 8	4
	2.2.3 Soil Description Equipment	5 of 8	4
	2.2.4 Photographic Equipment	6 of 8	4
	2.2.5 Clod Sampling Equipment.	6 of 8	4
	2.2.6 Sampling Equipment	7 of 8	4
	2.2.7 Transportation Equipment	7 of 8	4
	2.3 Use of Field Equipment.	8 of 8	4
3.0	SELECTION OF PEDON TO BE SAMPLED	1 of 7	4
	3.1 Identifying a Suitable Pedon for Sampling .	1 of 7	4
	3.2 Procedure for Locating a Suitable Pedon	3 of 7	
	3.3 Locating a Suitable Pedon of a Map Unit Inclusion	6 of 7	4
	3.4 Paired Pedons	7 of 7	4
4.0	PEDON EXCAVATION	1 of 4	4

DRAFT

TABLE OF CONTENTS (Continued)

Section		Page	Revision
	4.1 Standard Excavation	1 of 4	4
	4.1.1 Pit Size	1 of 4	4
	4.1.2 Steps in the Pit	2 of 4	4
	4.2 Excavation of Soils with Water Tables . . .	2 of 4	4
	4.3 Excavation of Organic Soils	4 of 4	4
	4.4 Soils Difficult to Excavate	4 of 4	4
5.0	SITE AND PROFILE DESCRIPTION	1 of 7	4
	5.1 Profile Preperation	2 of 7	4
	5.2 Photographs of Profile and Site	2 of 7	4
	5.3 Thick Horizons.	4 of 7	4
	5.4 Field Descriptions.	5 of 7	4
	5.5 Documents	6 of 7	4
6.0	FIELD SAMPLING PROCEDURES.	1 of 14	4
	6.1 Sampling the Pedon.	1 of 14	4
	6.1.1 Field Sampling Protocol.	1 of 14	4
	6.1.2 Important Points Concerning Soil Sampling	1 of 14	4
	6.2 Sample Size	2 of 14	4
	6.3 Sampling Procedure.	3 of 14	4
	6.3.1 Stratified Horizons	4 of 14	4
	6.3.2 Field Duplicates.	4 of 14	4
	6.4 Sampling Clods for Bulk-Density Determination	4 of 14	4
	6.4.1 Procedure.	5 of 14	4
	6.4.2 Transport of Clods	6 of 14	4
	6.5 Filling Sample Bag.	6 of 14	4
	6.6 NADSS Label A	8 of 14	4
	6.7 Delivery.	14 of 14	4

DRAFT

TABLE OF CONTENTS (Continued)

Section		Page	Revision
	PART III. PREPARATION LABORATORY		
7.0	PREPARATION LABORATORY PERSONNEL AND EQUIPMENT .	1 of 4	4
	7.1 Personnel	1 of 4	4
	7.2 Equipment	2 of 4	4
8.0	RECEIPT AND STORAGE OF SAMPLES	1 of 2	4
	8.1 Bulk Soil Samples	1 of 2	4
	8.2 Clods for Bulk Density.	2 of 2	4
9.0	SAMPLE PROCESSING.	1 of 13	4
	9.1 Air Drying.	1 of 13	4
	9.1.1 General Considerations	1 of 13	4
	9.1.2 Procedure.	2 of 13	4
	9.2 Crushing and Sieving.	3 of 13	4
	9.2.1 General Considerations	3 of 13	4
	9.2.2 Procedure	6 of 13	4
	9.2.3 Calculation of Percent Rock Fragments.	9 of 13	4
	9.3 Homogenization and Subsampling.	9 of 13	4
	9.3.1 General Considerations	9 of 13	4
	9.3.2 Procedure for Analytical Samples . .	10 of 13	4
	9.3.3 Procedure for Mineralogical Samples.	12 of 13	4
	9.4 Documentation	12 of 13	4
10.0	FORMATION AND SHIPPING OF BATCHES.	1 of 4	4
	10.1 Analytical Samples	1 of 4	4
	10.1.1 Procedure.	1 of 4	4
	10.2 Mineralogical Samples.	4 of 4	4

DRAFT

TABLE OF CONTENTS (Continued)

Section		Page	Revision
11.0	ANALYTICAL PROCEDURES.	1 of 11	4
	11.1 Rock fragments	1 of 11	4
	11.1.1 Procedure	1 of 11	4
	11.1.2 Calculations	2 of 11	4
	11.2 Moisture	2 of 11	4
	11.2.1 Procedure	2 of 11	4
	11.2.2 Calculations	4 of 11	4
	11.3 Inorganic Carbon	4 of 11	4
	11.3.1 Procedure	4 of 11	4
	11.3.2 Internal Quality Control	5 of 11	4
	11.4 Bulk Density	5 of 11	4
	11.4.1 Procedure	6 of 11	4
	11.4.2 Assumptions	9 of 11	4
	11.4.3 Calculations	9 of 11	4
12.0	REFERENCES	1 of 1	4

Appendices

A	Strategy of Site Selection and Sampling Information for the Northeastern United States	1 of 20	4
B	Strategy of Site Selection and Sampling Information for the Southeastern United States	1 of 4	4
C	Field Data Form and Legends.	1 of 42	4
D	Preparation Laboratory Forms	1 of 3	4
E	List of Northeast Soils by Sampling Class. . . .	1 of 12	4
F	List of Southern Blue Ridge Soils by Sampling Class.	1 of 12	4

DRAFT

ACKNOWLEDGMENTS

Contributions provided by the following individuals were greatly appreciated: S. Bodine, D. Lammers, M Johnson, J. Lee, B. Jordan, M. Mausbach, R. Nettleton, W. Lynn, F. Kaisacki, B. Waltman, W. Hanna, B. Rourke, G. Raab, and J. Warner.

The following people were instrumental in the timely completion of this manual: Computer Sciences Corporation word processing staff at the Environmental Monitoring Systems Laboratory-Las Vegas, C. Roberts at the Environmental Research Laboratory-Corvallis, J. Engels, M. Faber, and G. Villa at Lockheed Engineering and Management Services Company, Inc., and Mary Lou Putnam of Donald Clark Associates.

DRAFT

PART III. PREPARATION LABORATORY

7.0 PREPARATION LABORATORY PERSONNEL AND EQUIPMENT

7.1 Personnel

Personnel at the preparation laboratory will be responsible for distributing equipment and supplies provided by EMSL-LV to the field crews for maintaining an adequate inventory of supplies for the duration of the survey, and for returning all provided equipment and unused supplies to EMSL-LV at the close of the survey.

The preparation laboratory is also responsible for obtaining the acetone necessary for mixing the saran:acetone that is used by both the field crews and the preparation laboratory for the collection of clods and for the analysis of samples for bulk density.

The preparation laboratory is responsible for keeping complete documentation for the tracking of samples during storage and through the preparation procedures. The laboratory may be required to provide this documentation to the EPA project officer or designee during the course of the project. Required documentation must be submitted to the EPA upon conclusion of the survey.

DRAFT

The preparation laboratory is responsible for receipt of bulk soil samples and clods from the field crews and is also responsible for following the specified protocol for all aspects of storage, processing, subsampling, batching, and shipping of samples. The laboratory is responsible for performing the specified analytical procedures, i.e., bulk density, percent air-dry moisture, percent rock fragments, and the qualitative test for inorganic carbon and for keeping accurate and legible data books. The data books will be bound, kept in black ink, dated and signed by the analysts, with all erroneous entries initialed and crossed out so that they remain legible. The laboratory is responsible for reassignment of field samples to analytical batches and for keeping supporting documentation which includes the NADSS label A logbook and NADSS forms 101 and 102.

The manager of the preparation laboratory will be a person experienced in processing soil samples and organized in the tracking of samples through the laboratory.

7.2 Equipment

The preparation laboratory will be equipped to perform the specified protocol. A partial listing of equipment follows; items marked with an asterisk will be provided by EMSL-LV:

- Logbooks*

DRAFT

- Plastic and cloth sample bags*
- Twist ties for sample bags*
- Brown or white paper
- Drying trays
- Dehumidifier
- Wooden rolling pin
- Sieves, 2-mm mesh
  4.75-mm mesh
- Jones-type, riffle splitter with 1.25-cm openings
- Scale or two-pan balance
- NADSS labels A and B*
- NADSS forms 101 and 102*
- Pens, permanent ink*
- Bottles, 500-ml plastic*
- Shipping boxes*
- Packing material
- Strapping tape*
- Air courier account number*
- Porcelain spot plate
- Squeeze bottle or eyedropper for deionized water
- Microscope or stereoscope (10x or higher)
- 4 N HCl
- Quality control detection limit sample, i.e., soil spiked with 1 percent (wt/wt) $CaCO_3$ or $CaMg(CO_3)_2$
- Quality control calibration sample (QCCS) test soil spiked with 5% (wt/wt) $CaCO_3$

DRAFT

- QCCS test soil spiked with 5% (wt/wt) $CaMgCO_3$
- Top-loading balance
- Analytical balance
- Drying oven
- Muffle furnace
- Laboratory equipped with house air, fume hoods, safety equipment

DRAFT

8.0 RECEIPT AND STORAGE OF SAMPLES

8.1 Bulk Soil Samples

Samples are received at the preparation laboratory as soon as possible after sampling. Keep a logbook to record the following information: date received; time received; who delivered samples; who received samples; condition of samples, noting only problems specifically by sample code, e.g., if the samples were not held at 4°C prior to delivery or if a sample container broke and contamination is possible; set ID numbers; and total number of samples. The field crew is responsible for registering samples upon delivery unless laboratory personnel decide to accept this responsibility.

Unless the samples are taken immediately to the processing area and are spread out to air-dry, place the samples in cold storage upon receipt. Return samples to cold storage after drying and whenever they are not undergoing processing as described in Section 9.0. Maintain the cold storage locker at 4°C and monitor the temperature on a daily basis. Record any deviations in the temperature in a logbook and identify those samples affected by the change in temperature by set ID and, if necessary for clarity, by sample code.

Keep samples organized so that a particular sample may be located easily.

DRAFT

Shelving of some kind is necessary to maximize the use of available space and to facilitate organization of the samples.

8.2 Clods for Bulk Density

Clods are received at the preparation laboratory along with the bulk samples. A separate logbook is kept to record the receipt of clods.

Remove the label from each clod and staple it in the logbook. Immediately relabel the clod with the sample code and assign a replicate number to provide a unique identity for each clod. Also, record the date of receipt, the condition of each clod, the number of saran coatings, and the assigned replicate number.

DRAFT

9.0 SAMPLE PROCESSING

Specific areas will be designated for sample processing. During all steps of processing, sample integrity is of the greatest importance. This is protected by maintaining the unique identity of each sample by labeling with the sample code, by documenting each sample through all processing, and by avoiding physical and chemical contamination during each processing step.

9.1 Air Drying

9.1.1 General Considerations - The area designated for air-drying samples must have adequate bench space to allow several sets of samples, i.e., approximately 60 samples, to be drying simultaneously. Also, it is important that there be a method to reduce relative humidity in the room, e.g., by removing moist air via a fume hood or by reducing humidity with a dehumidifier. If available, a temperature- and humidity-controlled greenhouse environment is ideal for drying samples. The area or room must be secure and have limited access.

In evaluating an area for air-drying samples, sources of potential contamination, including growing plants, other sample types, dust, and chemicals such as fertilizers or pesticides, will be identified and eliminated. Handling samples without wearing gloves is a source of contamination as is the talc which is used on some plastic or rubber gloves.

DRAFT

Soils high in clay may harden irreversibly if allowed to dry completely prior to crushing; therefore, crush such soils before they reach a constant moisture content. Then continue air-drying after the crushing procedure (described in Section 9.2) and determine the moisture content after the air-drying is completed. It may be desirable to hold partially dry soils that are high in clay in cold storage until crushing may be scheduled.

9.1.2 Procedure

9.1.2.1 Spread the sample on a new sheet of white or brown paper large enough to allow paper to be exposed on all sides of the sample. The paper may be placed on a tray or directly upon the bench top.

9.1.2.2 Occasionally stir the soil with a clean stainless-steel spoon to facilitate drying. Water may condense on the tray or bench top, creating an undesirable situation that would maximize conditions for microbial growth. An arrangement allowing for air circulation below the sample is preferred. For example, frames with screen bottoms may be used in place of trays, or the bench top may be made of steel netting. As an alternative, place several layers of paper below the paper containing the sample and replace those layers when they become damp. Any observations of fungal or algal growth should be noted by sample code in the sample tracking log book.

DRAFT

9.1.2.3 Allow the sample to air-dry until it achieves a constant moisture content, i.e., until it is in equilibrium with the relative humidity. This step may require a period of two days or more than three weeks, depending upon the character of sample.

Test each sample for moisture content as described in Section 11.2. Constant air-dry moisture content is achieved when the moisture content is not reduced by more than an absolute 2.5 percent in two consecutive days. After the sample passes this test, it may undergo further processing immediately or be placed in a new plastic bag, sealed, and placed in cold storage until further processing is possible.

9.1.2.4 On rainy days or during periods of high relative humidity, a sample may adsorb moisture; therefore, determinations of moisture may show an increase in moisture content. Do not rebag a sample that has increased in moisture content because of such weather conditions. Allow additional, less humid days for it to dry. If necessary, replace the damp paper below the sample.

9.2 Crushing and Sieving

9.2.1 General Considerations - A fume hood with inspected and properly functioning fans, vents, and filters, is the preferred location for crushing and sieving samples. It is a relatively small area that can be covered with protective layers of paper and that can be cleaned easily. Also, the

DRAFT

exhaust removes small soil particles that would otherwise contaminate the work area and that would be inhaled by the technician.

An area near a source of compressed air is desirable because the air can be used to clean the surfaces of the equipment, i.e., wooden rolling pin, 4.75-mm mesh sieve, and 2-mm mesh sieve, after the processing of each sample. However, water and oil tend to accumulate in the conduit and to spray out occasionally from in-house, compressed-air lines. This would represent a source of contamination. Therefore, the air must be passed through a trap to collect the offending water and oil.

Such a trap can be assembled by using an Erlenmeyer flask, a small piece of cotton cloth or several Kimwipes, a one-holed rubber stopper sized to fit the flask, a piece of glass or plastic tubing long enough to extend through the rubber stopper and well past the side arm of the flask, and two lengths of vinyl tubing. Place the cloth or Kimwipes into the flask. Insert the glass or plastic tubing through the rubber stopper and stopper the flask. Connect the side arm of the flask to the air nozzle with a short length of vinyl tubing. The second length of vinyl tubing should be long enough to reach from the position of the flask to the work area. Attach it to the exposed end of the glass or plastic tubing which is in the rubber stopper. If water or oil is observed in this length of vinyl tubing when using air to clean the equipment, the cloth or Kimwipes should be replaced, and the tubing should be cleaned

DRAFT

out before continuing. Occasionally, the air pressure may be too high for this apparatus, and this will cause the rubber stopper to blow out. This problem can be corrected by adjusting the delivery of air from the nozzle.

In addition to cleaning the equipment with compressed air, the surfaces may be wiped with Kimwipes if soil is adhering to the wooden rolling pin or to the frame of the sieve. Used Kimwipes should be discarded after cleanup for each sample processed to avoid contamination. Sieves which have been cleaned with compressed air will be rinsed with deionized water and will be thoroughly air-dried or oven-dried at the end of each day. Brass sieves should be removed from the drying oven as soon as they are dry because brass will oxidize at temperatures as low as 60°C.

Large pieces of stems and roots that are obviously not part of the soil should be removed when encountered in the processing described in Section 9.2.2.

CAUTION: Rock fragments in soils of the Southeast are likely to be relatively soft either because they are of sedimentary origin or because they have been subjected to intensive weathering. Using a wooden rolling pin as specified in the procedure below will crush these soft rock fragments. This will result in an underestimation of percentage rock fragments in the laboratory

DRAFT

as compared to the field estimates and also will result in a sample of questionable integrity because it contains crushed rock material. When rock fragements are soft and easily crushed, the procedure may be modified according to the alternatives specified in the appropriate subsections below.

9.2.2 Procedure

9.2.2.1 Record the weight of the whole sample prior to crushing and sieving. Any sample lost during the processing is most likely to be from the less than 2-mm fraction.

9.2.2.2 Spread a small portion of sample on an approximately 60-cm by 60-cm piece of brown or white paper. Another layer of paper may be placed over the sample, if desired. Crush the sample with the rolling pin; apply only enough force to disaggregate the clods or soil structure, but not so much force that weathered rocks are crushed. Go to Section 9.2.2.3.

ALTERNATIVE: Place a portion of sample in the 2-mm sieve and gently push the soil through the sieve with a rubber stopper. Attempt to include soil adhering to the rock fragments. Continue until the entire sample is processed. Go to Section 9.2.2.5.

DRAFT

9.2.2.3 After each portion is crushed, set the portion aside and repeat with another aliquot until the entire sample is prepared. Go to Section 9.2.2.4. Alternatively, each portion may be sieved as described in Section 9.2.2.4 before continuing with another portion of sample.

9.2.2.4 Either nest the two sieves with the 2-mm mesh sieve on the bottom or use only the 2-mm mesh sieve for this step. Place the sieve(s) onto an approximately 45-cm by 45-cm piece of brown or white paper. Pour aliquots of soil onto the sieve(s). Move the sieve(s) from side to side, gently tapping the side of the sieve(s) to facilitate the passage of sample through the mesh. Do not press sample through the sieve(s). If peds are retained by the sieve(s), remove that material from the sieve(s) and crush as described in Section 9.2.2.3. Continue sieving until all material has been processed. Save the less than 2-mm material in plastic sample bags and return to cold storage or immediately continue with processing as described in Section 9.3.

9.2.2.5 If both the 4.75-mm and 2-mm sieves were used in Section 9.2.2.4, continue at Section 9.2.2.6.

If only the 2-mm mesh sieve was used in Section 9.2.2.4, sieve all rock material which was retained by the 2-mm mesh sieve through the 4.75-mm mesh sieve. Catch the material which passes the sieve on a piece of paper.

DRAFT

ALTERNATIVE: A significant amount of soil may adhere to the soft rock fragments. Wash the fragments with deionized water while they are on the sieve. Then continue with Section 9.2.2.6.

9.2.2.6 Weigh the material retained by the 4.75-mm sieve. Record the weight as that of the 20- to 4.75-mm rock fragments.

9.2.2.7 Weigh the material retained by the 2-mm sieve. Record the weight as that of the 4.75- to 2-mm rock fragments.

9.2.2.8 Combine the rock fragments in a plastic sample bag and label with the appropriate sample code.

If the qualitative test for inorganic carbon as described in Section 11.3 is positive, crush the rock fragments to pass a 2-mm mesh sieve. Use a Jones-type riffle splitter first to homogenize the sample and then to obtain a 100-g subsample. Crush the entire subsample to pass an 80-mesh sieve. Place it in a plastic sample bag labeled with the appropriate NADSS label. If crushing of rock fragments is done prior to sample batching, the 100-g subsample is labelled initially with NADSS Label A which is later replaced with NADSS Label B. If sample batching is done prior to the determination of inorganic carbon, the 100-g subsample is labeled with NADSS Label B. Place the plastic bag in a cloth sample bag. Keep this subsample with the appropriate 1-kg

DRAFT

analytical subsample for shipment to the analytical laboratory as described in Section 10.0.

Save all rock fragment samples until instructed to discard them or to ship them to another location. Cold storage is not required for rock fragments.

9.2.3 Calculation of Percent Rock Fragments - The percentage of rock fragments in each size faction, i.e., 20- to 4.75-mm and 4.75- to 2-mm, is calculated as follows:

Percent Rock Fragments in Size Fraction =

$$\left(\frac{[\text{weight of size fraction}]}{[\text{total weight}]}\right) 100$$

9.3 Homogenization and Subsampling

9.3.1 General Considerations - A fume hood is the preferred location for homogenization and subsampling the less than 2-mm fraction. It should be prepared as described in Section 9.2.1.

A Jones-type riffle splitter with 1.25-cm openings is used for both homogenization and subsampling. The riffle splitter will be cleaned between each sample with compressed air as described in Section 9.2.1. The surfaces may be cleaned with a brush, if necessary.

DRAFT

It will be cleaned with deionized water and allowed to thoroughly air-dry after each day of use. Prior to use, the riffle spitter should be inspected to verify that it is dry.

A scale or two-pan balance must be available to weigh the 1-kg analytical sample or the 500-g mineralogical sample.

Because (1) only a few horizons will be chosen for mineralogical analyses from all routine samples collected and (2) the identification of those horizons is made after all samples have been collected, samples for mineralogical analyses will be split at some time after those for analytical analyses are split.

9.3.2 Procedure for Analytical Samples

9.3.2.1 Position the receiving pans on each side of the riffle splitter. Pour the less than 2-mm material evenly across the baffles of the riffle splitter. Repeat six times in succession for the material in each receiving pan to ensure that the sample is thoroughly homogenized.

9.3.2.2 Transfer the entire sample to one receiving pan or pour it onto a new square of brown or white paper.

Place the receiving pans on each side of the riffle splitter. Pour the sample evenly across the baffles. Place the material from one

DRAFT

receiving pan into a plastic sample bag. With the material in the other receiving pan, repeat the procedure as required until a subsample of approximately 1 kg is obtained.

It is important that the subsample be obtained entirely by splitting with the riffle splitter. An alternate method of using the riffle splitter to obtain the 1-kg subsample follows: After an easily handled amount of sample is split out, a subsample of 1 kg may be obtained by splitting the material and allowing the material to remain in both pans. Then the material from one pan is passed through the splitter again, so that one pan now contains 3/4 of the material and the other pan contains 1/4 of the material. Place the smaller amount of material in the sample bag and continue to split in this manner until the 1-kg sample is obtained. If only a small amount of material is needed to attain the 1-kg subsample, successive splits of the material may be made so that the pan containing the lesser amount of material contains 1/8, 1/16, 1/32, and so on, and the pan containing the greater portion of material contains 7/8, 15/16, 31/32, and so on.

9.3.2.3 Place the 1-kg subsample in a plastic sample bag and secure with a twist tie. Label with NADSS Label A or, if an analytical batch is being assembled as described in Section 10.0, NADSS Label B may be placed on the 1-kg subsample. The sample may be double-bagged in

DRAFT

plastic for security. Place the plastic bag in a cloth bag and return the subsample to cold storage as soon as possible.

9.3.2.4 Label remaining sample material with NADSS Label A. Place the plastic bag in a cloth bag and return the sample to cold storage for archiving. Sets will be packed according to sample code for permanent archiving.

9.3.3 Procedure for Mineralogical Samples

9.3.3.1 Rehomogenize the less than 2-mm material as described in Section 9.3.2.1.

9.3.3.2 Split out a 500-g subsample as described in Section 9.3.2.2.

9.3.3.3 Place the 500-g subsample in a 500-mL air-tight plastic bottle. Label the bottle with the sample code and return the sample to cold storage until it is shipped.

9.4 Documentation

In addition to data books used for entry of raw data for determination of percent moisture and percent rock fragments, records will be kept to document the date(s) of sample processing.

DRAFT

Also, the weight of the less than 2-mm material obtained after sieving, the percent rock fragments in the 20- to 4.75-mm fraction, and the percent rock fragments in the 4.75- to 2-mm fraction will be entered into the NADSS Label A logbook described in Section 10.1.1.4.

DRAFT

10.0 FORMATION AND SHIPPING OF BATCHES

10.1 Analytical Samples

A batch of samples for chemical and physical analysis by a contractor laboratory consists of a maximum of 39 routine samples and field duplicates. Depending upon the number of samples in each set, i.e., a group of samples taken by one field crew in 1 day, a batch may include up to six sets. In addition, one sample per batch is chosen as the preparation duplicate and is split into two subsamples according to the procedure in Section 9.4. Two audit samples supplied by the quality assurance staff will be specified for each batch. Therefore, one batch including routine samples, field duplicates, a preparation duplicate, and two audit samples will contain a maximum of 42 samples.

10.1.1 Procedure

10.1.1.1 After deciding which sets will be included in the analytical batch, randomly choose one sample to be the preparation duplicate. The horizon-type chosen as the preparation duplicate should vary from batch to batch so that all horizon-types are chosen at least once. Rehomogenize the archived sample material for the chosen sample by seven successive passes through a Jones-type riffle splitter. Then split out a subsample as described in Section 9.4.

DRAFT

10.1.1.2 Randomize the samples in the batch. Assign sample numbers from 1 through 42, as needed. Record the site ID, sample code, and set ID on NADSS Form 101.

The sample code for a preparation duplicate will begin with three alpha characters, PLD, and end with the same last 10 digits as the routine sample from which it was split. For example, the preparation duplicate split from routine sample R11NH01300405 would be designated as PLDNH01300405.

The sample code for an audit sample would begin with the first three characters of the horizon designation and would be followed by two zeros, the three-digit audit number specified by the quality assurance staff, a hyphen, and four zeros. For example, the audit sample prepared from soil sampled from an argillic, maximum B horizon would begin with the three characters, B+2. If the horizon designation includes less than three characters, the spaces are filled in with zeros.

Set NADSS Form 101 aside until the remaining data for weight percent rock fragments, weight percent air-dry moisture, soil type, presence of inorganic carbon, and bulk density may be entered from the appropriate data books. For sample tracking purposes, it is important that this form be sent as soon as possible. After it is completed,

DRAFT

Form 101 will be submitted to the following address:

Lockheed Engineering and Management Services Company, Inc.
1050 East Flamingo, Suite 200
Las Vegas, Nevada 89109

10.1.1.3 Fill out NADSS Label B for each sample. This label includes only the batch number and the sample number assigned above.

10.1.1.4 Remove NADSS Label A from each sample and replace it with NADSS Label B. Staple NADSS Label A in a logbook and initial the label so that the writing overlaps both the label and the page. Indicate in the logbook the newly assigned batch and sample numbers.

10.1.1.5 Fill out NADSS Form 102. Remove the white (original) copy and send it via overnight courier to the Sample Management Office at the following address:

Sample Management Office (Viar)
300 North Lee Street
Alexandria, Virginia 22314

The remaining copies are left intact and are included inside the packed box(es).

10.1.1.6 Each sample should now be labeled with only the newly assigned batch ID and sample number. Place each plastic bag of sample in a cloth

DRAFT

bag. Secure the cloth bag so that any sample that might leak from the plastic bag will be contained by the cloth bag. Pack the samples securely, by using additional packing materials as needed.

10.1.1.7 Store the packed boxes under refrigeration at 4°C until they are to be shipped.

10.1.1.8 Send the box(es) containing the designated analytical batch to the contractor laboratory specified by the quality assurance staff. An overnight air courier service will be specified for all shipments, and an account number will be supplied for billing shipments to a third party.

10.2 Mineralogical Samples

Samples for mineralogical analysis will be combined into batches of 20 routine samples, 3 preparation duplicates, and 3 audit samples at EMSL-LV. The subsamples will be shipped via overnight air courier to the following address where they will be combined into batches and forwarded to the designated mineralogical laboratory:

Lockheed Engineering and Management Services Company, Inc.
1050 East Flamingo Road, Suite 200
Las Vegas, Nevada 89109

DRAFT

11.0 ANALYTICAL PROCEDURES

11.1 Rock Fragments

The term "rock fragments" is defined in Soil Taxonomy as "particles 2 mm in diameter or larger and includes all sizes that have horizontal dimensions less than the size of a pedon." Only the 20- to 4.75-mm fraction and the 4.75- to 2-mm fraction are determined in the preparation laboratory. The percentage of rock fragments is determined on the basis of the weight of sample brought into the laboratory for processing.

11.1.1 Procedure

11.1.1.1 Record the weight of the whole sample prior to crushing and sieving. Any sample lost during the processing described in Section 9.2 is most likely to be from the less than 2-mm fraction.

11.1.1.2 Weigh the material retained by the 4.75-mm sieve. Record the weight as that of the 20- to 4.75-mm rock fragments.

11.1.1.3 Weigh the material retained by the 2-mm sieve. Record the weight as that of the 4.75- to 2-mm rock fragments.

DRAFT

11.1.2 Calculations - The percentage of rock fragments in each size faction, i.e., 20- to 4.75-mm and 4.75- to 2-mm, is calculated as follows:

$$\text{Percent Rock Fragments in Size Fraction} = \left(\frac{[\text{weight of size fraction}]}{[\text{total weight}]}\right) 100$$

11.2 Moisture

Moisture content is determined on a 15- to 20-g grab sample taken after the air-dry sample described in Section 9.1.2.2 has been thoroughly mixed. Two convection-type drying ovens are preferred for this procedure if both mineral and organic soils are tested because mineral soils are dried at $\pm$ 5°C and organic soils are dried at 60°C $\pm$ 5°C. Each oven must equilibrate at the appropriate temperature for 24 hours prior to use. If only one drying oven is available, allow 24 hours for the temperature to equilibrate after an adjustment is made from one temperature to the other. The range of the thermometers used to measure oven temperature will be 0 to 200°C.

11.2.1 Procedure

11.2.1.1 Thoroughly mix the air-dried soil. Transfer a 15- to 20-g sample to an aluminum weighing dish which is not to exceed 2 g in weight. Handle the weighing dish with forceps or finger cots.

DRAFT

11.2.1.2 Record the initial weight of sample and weighing dish to the nearest 0.01 g. Because the aluminum weighing dishes are manufactured to be a nearly-consistent weight, the average weight of 10 aluminum weighing dishes may be used as the tare weight.

11.2.1.3 Dry the sample overnight or for 16 hours in a drying oven which is equilibrated at the appropriate temperature.

11.2.1.4 Remove the sample from the oven and allow it to cool for at least 30 minutes in a desiccator. Then record the oven-dried weight of the sample and weighing dish.

11.2.1.5 On the following day, transfer a second 15- to 20-g sample from the central portion of the air-dried material to an aluminum weighing dish. Repeat steps 11.2.1.2 through 11.2.1.4.

11.2.1.6 If the calculated moisture contents differ by more than an absolute 2.5 percent, allow the sample to continue air-drying and retest it at a later time. However, if the calculated moisture contents do not differ by more than 2.5 percent in two consecutive days, the air-dry sample may undergo processing as described in Section 9.0.

DRAFT

11.2.2 Calculations

$$\text{Percent moisture} = \left(\frac{[\text{air-dried weight} - \text{oven-dried weight}]}{[\text{oven-dried weight}]}\right) 100$$

11.3 Inorganic Carbon

The less than 2-mm fraction is tested qualitatively for the presence of carbonate minerals, i.e., inorganic carbon. If carbonate minerals are present in the soil at levels of approximately 1 percent or higher, effervescence is observed after the addition of 4 N HCl.

When a sample is found to contain inorganic carbon by using this test, both the less than 2-mm material and the 20- to 2-mm rock fragments are submitted to the analytical laboratory for analysis. Refer to Section 9.2.2.8.

11.3.1 Procedure

11.3.1.1 Place 1 g of less than 2-mm soil material in a well of a porcelain spot plate. Thoroughly moisten the soil with a few drops of deionized water; stir with a clean glass rod to remove entrapped air.

DRAFT

11.3.1.2 Add three drops of 4 N HCl and immediately observe the treated sample under a light microscope or stereoscope. Be careful not to get acid on lens or lens sealant. If this occurs, wash off acid and clean and dry lenses. Record presence or absence of effervescence for each sample according to sample code.

11.3.1.3 Repeat the treatment and the observation with a second 1-g sample.

11.3.2 Internal Quality Control

Both a quality control (QC) detection limit sample and a quality control calibration sample (QCCS) are required for this procedure. The purpose of the QC samples is to ensure that the technician is able to distinguish effervescence.

The QC detection limit sample is prepared by spiking an aliquot of non-carbonate soil with 1 percent (wt/wt) $CaCO_3$ or $CaMg(CO_3)_2$. The QC detection limit sample is analyzed prior to sample analysis and after each 10 or fewer samples.

The QCCS is prepared by spiking an aliquot of non-carbonate soil with 5 percent (wt/wt) $CaCO_3$ or $CaMg(CO_3)_2$. The QCCS is analyzed prior to sample analysis and after each twenty or fewer samples.

11.4 Bulk Density

DRAFT

Density is defined as mass per unit volume expressed in units of g/cm^3. The bulk density of a soil is defined as the mass of dry soil per unit volume including the pore space. The bulk volume is determined prior to drying the soil to constant weight, at 105°C for mineral soils and at 60°C for organic soils.

Bulk density of mineral soils generally ranges between 1.0 and 2.0 g/cm^3. Soils that are porous will have low bulk density values, and soils that are compacted will have high bulk density values. With increasing organic matter content, soils generally exhibit a decrease in bulk density because (1) organic matter is less dense than mineral particles of the same size and (2) organic matter promotes granular soil structure with a resulting increase in porosity.

11.4.1 Procedure

11.4.1.1 Weigh the clod as received from the field crew. Record the initial weight as m_1. Clods weighing more than 100 g are weighed to the nearest 0.1 g; clods weighing less than 100 g are weighed to the nearest 0.01 g.

11.4.1.2 Briefly dip the clod into a 1:7 (wt/wt) saran-acetone mixture. Then suspend the clod from a line and allow the clod to dry.

DRAFT

11.4.1.3 Reweigh the clod and record this weight as m_2.

11.4.1.4 Repeat steps 11.4.1.2 and 11.4.1.3 as necessary until a coating of saran that is impervious to water is obtained. Record each additional weight as m_3, m_4,..., m_i.

11.4.1.5 Degas approximately 800 mL of deionized water in a 1-L beaker by boiling until no rising gas bubbles are observed. Cover the beaker with a watch glass and allow the water to cool to room temperature.

11.4.1.6 Place the beaker of degassed water on a balance and record the tare weight. Also, record the temperature of the water so that the density of the water may be obtained from Table 11.1.

11.4.1.7 Suspend the clod over the beaker. Lower the clod gently into the water until it is totally submerged. Record the weight displayed on the balance as weight of water supporting clod.

11.4.1.8 Suspend the clod in a drying oven at the appropriate temperature for 48 hours: 105°C for mineral soils and 60°C for organic soils. Moisture from within the clod will diffuse through the saran coating.

11.4.1.9 Remove the clod from the oven and allow it to cool in a desiccator. Weigh the clod to obtain the oven-dried weight.

DRAFT

TABLE 11.1. DENSITY* OF WATER

°C	0	1	2	3	4	5	6	7	8	9
0	0.9999	0.9999	1.0000	1.0000	1.0000	1.0000	1.0000	0.9999	0.9999	0.9999
10	0.9997	0.9996	0.9995	0.9994	0.9993	0.9991	0.9990	0.9988	0.9986	0.9984
20	0.9982	0.9980	0.9978	0.9976	0.99973	0.9971	0.9968	0.9965	0.9963	0.9960
30	0.9957	0.9954	0.9951	0.9947	0.9944	0.9941	0.9937	0.9934	0.9930	0.9926
40	0.9922	0.9919	0.9915	0.9911	0.9907	0.9902	0.9898	0.9894	0.9890	0.9885
50	0.9881	0.9876	0.9872	0.9867	0.9862	0.9857	0.9852	0.9848	0.9842	0.9838
60	0.9832	0.9827	0.9822	0.9817	0.9811	0.9806	0.9800	0.9795	0.9789	0.9784
70	0.9778	0.9772	0.9767	0.9761	0.9755	0.9749	0.9743	0.9737	0.9731	0.9724
80	0.9718	0.9712	0.9706	0.9699	0.9693	0.9686	0.9680	0.9673	0.9667	0.9660
90	0.9653	0.9647	0.9640	0.9633	0.9626	0.9619	0.9612	0.9605	0.9598	0.9591

*Also the specific gravity or unit weight of water in grams per milliliter.

DRAFT

11.4.1.10 For mineral soils, place the clod in a container that will withstand 400°C. Place the clod and container in a muffle furnace that is equilibrated at 400°C and allow the saran to vaporize from the clod over the next 2 hours. The muffle furnace must be placed in a fume hood: saran is a carcinogen.

Remove the container and contents from the muffle furnace and allow to cool in a desiccator. Weigh only the contents of the container. Record the weight of the soil particles.

11.4.1.11 Pass the sample through a 2-mm mesh sieve. Weigh both the rock fragments and the less than 2-mm fine earth fraction. Record the weights as m_{rf} and m_{ff}, respectively.

11.4.2 Assumptions - The following assumptions are made in this procedure:

- The weight of each field-applied saran coating is equivalent to each coating applied in the laboratory.
- Saran has not infiltrated the clod.
- The specific gravity of air-dried saran is 1.30 g/cm^3.
- Saran loses 15 percent of its weight upon oven-drying for 48 hours.
- The density of the rock fragments is 2.65 g/cm^3.

11.4.3 Calculations

DRAFT

11.4.3.1 Calculate the weight of the saran coatings before oven-drying:

$$\text{Weight of saran} = \left(\frac{[\text{number of coats (final clod wt - initial clod wt)}]}{[(\text{number of coats in lab}) - 1]}\right)$$

11.4.3.2 Calculate the weight of the saran coating after oven-drying:

$$\text{OD Weight of saran} = (0.85)\ (\text{Weight of saran})$$

11.4.3.3 Calculate the volume of the saran coatings:

$$\text{Saran vol} = \left(\frac{\text{Wt of saran}}{1.30}\right)$$

11.4.3.4 Calculate the volume of water displaced by the clod:

$$\text{Vol of water} = \left(\frac{\text{Wt of water}}{(\text{density of water})}\right)$$

11.4.3.5 Calculate the volume of rock fragments in the clod:

$$\text{Vol of RF} = \left(\frac{\text{Wt of RF}}{2.65}\right)$$

DRAFT

11.4.3.6 Calculate the volume of the less than 2-mm fraction and pore space:

$$\text{Vol of fines and pores} = [\text{Vol of water} - (\text{Vol of RF} + \text{Vol of saran})]$$

11.4.3.7 Finally, calculate the field moist bulk density:

$$\text{Bulk Density} = \left(\frac{[\text{OD clod wt} - (\text{wt of RF} + \text{OD saran wt})]}{\text{Vol of fines and pores}}\right)$$

DRAFT

12.0 REFERENCES

1. USDA/SCS. 1983. National Soils Handbook. Part 600-606. U.S. Government Printing Office, Washington D.C.

2. USDA/SCS. 1984. SCS National Soil Survey Manual. U.S. Government Printing Office, Washington D.C.

3. Mausbach, M., R. Yeck, D. Nettleton, and W. Lynn. 1983. Principles and Procedures for Using Soil Survey Laboratory Data. National Soil Survey Laboratory. Lincoln, Nebraska.

4. USDA/SCS. 1981. National Handbook of Plant Names. U.S. Government Printing Office, Washington, D.C.

5. USDA/SCS. 1984b. Soil Survey Laboratory Methods and Procedures for Collecting Soil Samples. Soil Survey Investigations Report No. 1. U.S. Government Printing Office, Washington D.C.

DRAFT